

CLAIMS

1 - Process for preparation of a nitrophenol from a nitrohalobenzene which consists in performing:

5 - (a) hydrolysis of a nitrohalobenzene compound by reaction of the said compound with a base,

- (b) acidification to obtain the nitrophenol compound from its salt, by an acid treatment,

- (c) crystallization of the nitrophenol compound obtained,

- (d) separation of the product obtained,

characterized in that it also includes at least the following steps:

15 - (e) concentration of the reaction medium after hydrolysis (a) and before acidification (b),

- (f) liquid/liquid decantation performed after acidification (b) and before crystallization (c) and intended to remove the aqueous phase obtained after acidification (b).

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2 - Process according to Claim 1, characterized in that the process comprises the following steps of hydrolysis of the nitrohalobenzene compound, concentration of the reaction medium, acidification, decantation,

25 crystallization of the nitrophenol and separation.

30 3 - Process according to Claim 1, characterized in that the process comprises the following steps of hydrolysis of the nitrohalobenzene compound, concentration of the reaction medium, crystallization of the nitrophenate, separation, acidification, decantation, crystallization of the nitrophenol and separation.

35 4 - Process according to Claim 1, characterized in that the process comprises the following steps of hydrolysis of the nitrohalobenzene compound, concentration of the reaction medium, optionally crystallization of the nitrophenate followed by separation thereof,

acidification, decantation, washing of the organic phase, crystallization of the nitrophenol and separation.

5 5 - Process according to one of Claims 1 to 4, characterized in that the basic hydrolysis of the nitrohalobenzene compound is effected by reacting it with an inorganic or organic base, preferably sodium or potassium hydroxide.

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6 - Process according to Claim 5, characterized in that the hydrolysis temperature lies between 100°C and 200°C, preferably between 140°C and 180°C.

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7 - Process according to Claim 1, characterized in that concentration of the reaction medium is performed so as to increase the concentration of nitrophenol in the medium from 0.1% by weight to 10% by weight, preferably from 0.5% to 3%.

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8 - Process according to Claim 7, characterized in that the concentration is increased by decreasing the reaction pressure, by pressure release, while remaining in the aforesaid temperature zone or by distilling 25 under atmospheric pressure at a temperature of the order of 100°C, under a pressure slightly lower than atmospheric pressure selected so as to have a distillation temperature lying between 80°C and 99.6°C or at a pressure greater than atmospheric pressure.

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9 - Process according to one of Claims 1 to 8, characterized in that the acidification is performed by addition of a protonic acid of inorganic origin, preferably hydrochloric acid or sulfuric acid.

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10 - Process according to Claim 9, characterized in that the quantity of acid is at least equal to the quantity necessary for obtaining a pH lying between 1

and 7, preferably between 2 and 5, at the end of acidification.

5 11 - Process according to Claim 9, characterized in that the reaction medium is maintained at a temperature varying between 45°C and 70°C and preferably from 50°C to 60°C.

10 12 - Process according to one of Claims 1 to 11, characterized in that the crystallization of the nitrophenol is performed by cooling to a temperature which is a temperature lower than 40°C, preferably the ambient temperature or even lower.

15 13 - Process according to one of Claims 1 to 12, characterized in that the separation of the crystallized product is performed by the standard techniques of solid/liquid separation, preferably by filtration or by centrifugation.

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14 - Process according to Claim 1, characterized in that an operation of decantation of the two liquid phases obtained is performed following the acidification, in the range of temperatures stated 25 above for the acidification, preferably 60 - 70°C.

15 - Process according to one of Claims 3 and 4, characterized in that crystallization of the nitrophenate is performed at the end of the 30 concentration operation, by cooling to an ambient temperature and that the crystallized product is separated by the standard techniques of solid/liquid separation, preferably by filtration or centrifugation.

35 16 - Process according to one of Claims 2, 3 and 4, characterized in that a step of water washing of the organic phase is interposed between the decantation and the crystallization.

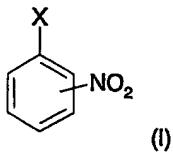
17 - Process according to Claim 16, characterized in
 that the mother liquors and washings from the
 crystallization of the nitrophenol are recycled to the
 5 hydrolysis of the nitrohalobenzene or to the dilution
 of the crystallized phenate after acidification.

18 - Process according to Claim 16, characterized in
 that at least part of the decantation liquors which
 10 result the the acidification of the nitrophenate is
 recycled to the hydrolysis of the nitrohalobenzene or
 to the dilution of the crystallized phenate after
 acidification.

15 19 - Process according to Claim 16, characterized in
 that the solid which precipitates to part of the cooled
 decantation liquors is recycled to the hydrolysis of
 the nitrohalobenzene or to the dilution of the
 crystallized phenate after acidification.
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20 - Process according to Claim 4, characterized in
 that the aqueous washings of the organic phase which
 decanted after acidification of the nitrophenate are
 recycled to the hydrolysis of the nitrohalobenzene or
 25 to the dilution of the crystallized phenate after
 acidification.

21 - Process according to one of Claims 1 to 20,
 characterized in that the nitrohalobenzene corresponds
 30 to the formula:



(I)

in the said formula (I):

- X represents a fluorine, chlorine, bromine or iodine atom, preferably a chlorine atom (I),
- 35 - the NO₂ group is in the ortho, meta or para position, and preferably in the para position.

22 - Process according to Claim 21, characterized in
that the nitrohalobenzene corresponding to the formula
(I) bears one or several other halogen atom(s) or one
5 or several nitro group(s) or one or several alkyl
group(s) having from 1 to 4 carbon atoms.

23 - Process according to Claim 21, characterized in
that the nitrohalobenzene is p-nitro-chlorobenzene.

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24 - Nitrophenol having:

- a nitrohalobenzene content less than 180 ppm,
preferably less than 50 ppm.
- a halogen ions content less than 40 ppm,
15 preferably less than 20 ppm.

25 - Nitrophenol according to Claim 24, characterized
in that it has a sulfur content preferably lower than
200 ppm, and still more preferably lower 100 ppm.

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26 - p-Nitrophenol having:

- a p-nitrohalobenzene content less than 180
ppm, preferably less than 50 ppm.
- a halogen ions content less than 40 ppm,
25 preferably less than 20 ppm.

27 - p-Nitrophenol according to Claim 26, characterized
in that it has a sulfur content preferably lower than
200 ppm, and still more preferably lower 100 ppm.

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